UNIVERSITY OF CALIFORNIA
COLLEGE OF AGRICULTURE
AGRICULTURAL EXPERIMENT STATION
BERKELEY, CALIFORNIA

CIRCULAR 340
APRIL, 1937

TESTING MILK AND CREAM[1]

D. H. NELSON[2]

This circular describes the approved procedure for the simplest, easiest tests for the quality of milk and cream. With the directions here given the tests should be successfully performed by persons not familiar with milk testing or with laboratory technique. Such persons, however, may well observe first the work of an experienced laboratory technician. The highest accuracy can be attained only by skilled operators with very careful attention given to all details of the procedure.

CLEANING THE EQUIPMENT

Accurate results and successful operation depend upon using equipment that is completely cleaned. Since the procedure in cleaning testing equipment is the same for all tests, it is described first. The equipment should be washed immediately after use, before it dries. The first step is to rinse it thoroughly with clean water having a temperature between 90° and 100° Fahrenheit. Then follows a thorough brushing in a solution of some alkali such as sal soda, lye, or some suitable commercial cleaning preparation made for that purpose. About a tablespoonful of powder in 6 or 8 quarts of water usually suffices. The temperature of this solution should be between 110° and 120°—not too hot to be comfortable for the hands. Then the equipment should be rinsed at least twice with clean water at about the same temperature as that used for washing. This procedure should be followed in washing all equipment, such as sample bottle, pipettes, and test bottles.

[1] This publication supersedes Circular 230, *Testing Milk, Cream, and Skim Milk for Butterfat*, by J. C. Marquardt, and its later revisions.

[2] Associate in Dairy Industry.

SAMPLING[3]

Sampling is the first important step in any test: an inaccurate sample will of course give incorrect results regardless of the accuracy of the testing. The experience and skill of the operator cannot offset inaccurate sampling. The most satisfactory method of mixing milk for sampling is to pour it from one container to another. If the cream has risen one must repeat the pouring three or four times and perhaps warm the milk slightly in order to soften the lumps of cream and to distribute the fat uniformly throughout the milk. In cow-testing work the complete milking is poured from one pail to another as soon as it is drawn, and the sample taken at once. The sample must be taken immediately after mixing, because the fat soon begins to rise.

Milk stirrers or mechanical agitators should be used if the milk cannot be poured from one container to another. Great care is needed to insure thorough mixing immediately before the sample is taken.

One should take at least two fluid ounces of milk for a sample in order that retests may be made in case of error. If the sample is to be shipped or carried about, the bottle should be completely filled in order to prevent partial churning by the agitation. The sample should be placed in a clean bottle, tightly stoppered to prevent leakage or evaporation. The sample bottle should be so prepared that the label will not be easily removed or confused in handling. Samples should be kept in a cool place out of direct sunlight. Sampling equipment commonly used is shown in figure 1.

Composite Samples.—A composite sample is a mixture of representative samples from two or more lots of milk, taken in proportion to the amount of milk in each lot. In official testing, composite samples are taken with a graduated pipette; and a certain quantity of milk, usually 3 milliliters (ml),[4] is taken for each pound of milk produced at each milking. Sampling tubes are sometimes used; but they will give proportional samples only when the milk containers have perpendicular sides, flat bottoms, and equal diameters or areas. An accurate composite sample will test the same as the average test, which is computed from the tests and weights of the individual lots of milk. A preservative (corrosive sublimate, formaldehyde, or potassium dichromate) should be added to composite samples if they are to be collected over a period of days. Pre-

[3] These directions also apply to cream; but the viscosity of cream makes a uniform mixture more difficult to obtain.

[4] For practical purposes a cubic centimeter (cc) may be considered equivalent to a milliliter. Since the manufacturers of dairy equipment are now marking their glassware in milliliters this standard of measurement is used throughout this publication.

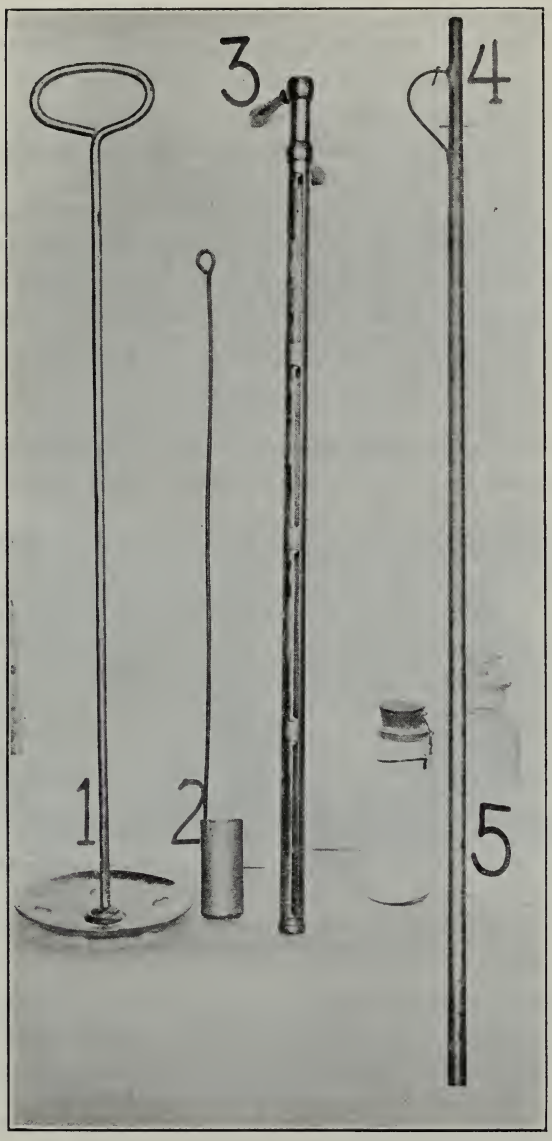

Fig. 1.—Sampling equipment: *1*, type of rod commonly used for stirring milk; *2*, dipper type of sampler now in general use for taking milk samples; *3*, the McKay sampler occasionally used for milk sampling but designed for cream sampling; *4*, thief sampler, efficiently used for milk sampling; *5*, two types of bottles satisfactory for preserving composite samples. (From Cir. 230.)

servatives purchased from dairy supply houses are usually accompanied by directions for their use.

Churned Samples.—Samples containing butter particles ("churned samples") are unsatisfactory for testing. If, however, a test must be made, warm the sample to 110° F and maintain this temperature until all the lumps of fat are melted. Then mix and shake violently, taking the sample quickly before the melted fat has time to rise.

Curdled Samples.—Curdled samples should never be tested in official work; but when such testing is necessary, add sufficient powdered caustic soda or potash to dissolve the coagulated substance. Then make the test in the usual manner. As the volume of the sample is not changed, direct measurement of the fat column gives the correct reading.

FAT IN MILK

The fat content may be easily determined by the Babcock test, invented by Dr. S. M. Babcock in 1890 at the Wisconsin Agricultural Experiment

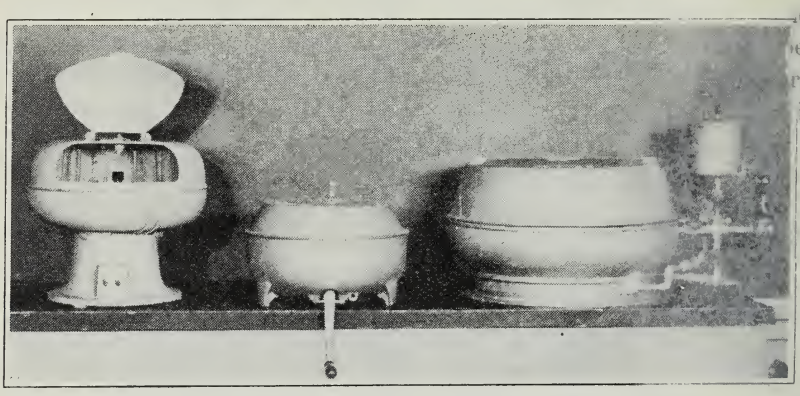

Fig. 2.—Left, electric centrifuge; center, hand centrifuge; right, steam-turbine centrifuge.

Station and since adopted throughout this country. It is based, first, on the use of strong sulfuric acid to dissolve the solids-not-fat and produce heat that aids in coalescing the fat globules and, second, on the use of a centrifuge to separate the fat completely from the acid mixture, which has a higher specific gravity.

The Acid.—The acid is described as commercial sulfuric acid, water-clear, and strong enough to have a specific gravity of between 1.82 and 1.83. Its corrosive action makes it extremely dangerous to handle. It attacks glass, lead, and oil or fat very slowly but most other substances very rapidly. When spilled the acid should be immediately washed away with large quantities of cold water, and its corrosive action checked with

a dilute alkali such as ammonia or an alkali washing solution. Sulfuric acid should be kept tightly stoppered at all times; otherwise it may absorb moisture from the air and become too weak for use.

The acidimeter illustrated in figure 3, *E* is the instrument commonly used for determining the strength of commercial sulfuric acid. It is standardized to read directly the specific gravity at 60° F.

The Centrifuge.—The centrifuge or tester may be turned by hand, by electric motor, or by steam. These three types of centrifuges are shown in figure 2. The steam centrifuge is preferable because with it one can easily maintain a uniform speed and a uniform temperature in the machine. In every case the speed of the centrifuge is very important and depends upon the diameter of the socket carrier. The diameter is measured from the bottom of the sockets on one side to the bottom of the sockets directly opposite, when the sockets are in a horizontal position. The correct speeds for testers of various diameters are given below:

Diameter of socket carrier	Revolutions per minute of socket carrier
14 inches	875–925
16 inches	825–875
18 inches	775–825
20 inches	725–775
21 inches	710–760
22 inches	700–750
24 inches	675–725

In addition, the centrifuge must be set on a level and solid foundation to which it is securely fastened.

Milk Test Bottles.[5]—The only milk test bottle legal in California is graduated on the neck to read from 0 to 8 per cent when 18 grams of milk (delivered by the 17.6 ml pipette) is used for the test. Each of the smaller divisions represents 0.1 per cent. Such a test bottle is shown in figure 3, *B*.

The Milk Pipette.[6]—The pipette (fig. 3, *D*) should be graduated to contain 17.6 ml of water at 20° centigrade and should have a delivery tube small enough to be easily slipped into the neck of the test bottle and long enough to extend through the neck.

Procedure in Testing Milk for Fat.—The sample should be prepared by adjusting its temperature between 60° and 70° F and then thoroughly mixed by pouring from one container to another at least three or four times. The milk should be poured down the side of the container to

[5] For official work, as in ordinary creamery practice, glassware used in testing should bear the letters D. B., signifying that it has been found by the California State Department of Agriculture to be accurately graduated.

[6] See footnote 5.

avoid partial churning of the fat. If lumps of cream do not entirely disappear, the sample must be warmed to a temperature not exceeding 100° and mixed; but in such cases it must be cooled to between 60° and 70° before pipetting.

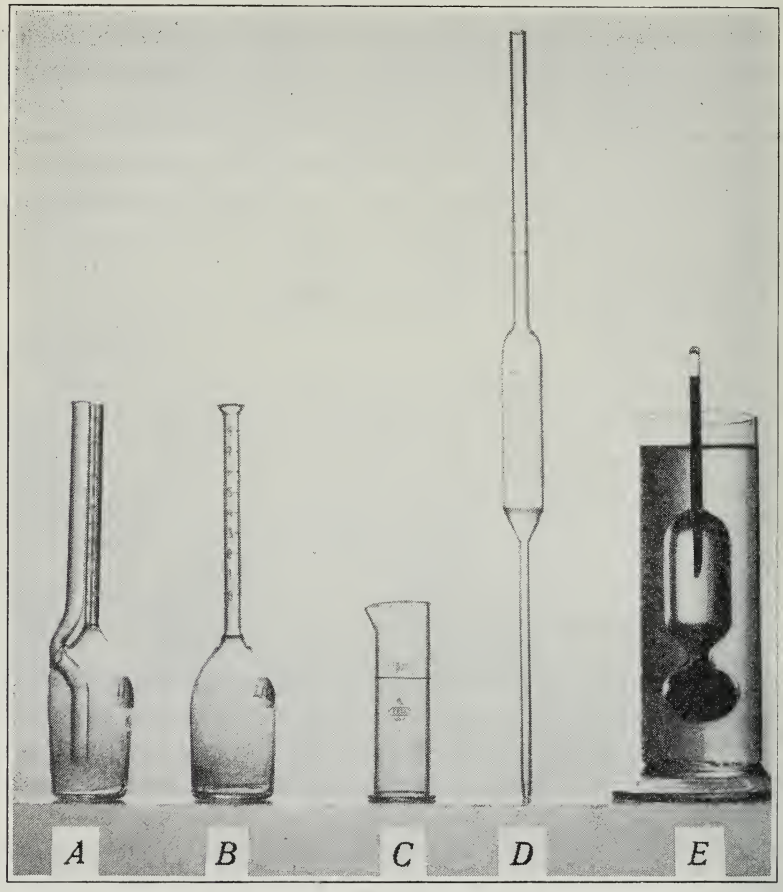

Fig. 3.—Glassware for testing: *A*, skim milk test bottle, 0.5 per cent; *B*, milk test bottle, 8 per cent; *C*, acid measure, 17.5 milliliters capacity; *D*, milk pipette, 17.6 milliliters capacity; *E*, acidimeter.

Immediately after preparing the sample, the operator sucks the milk into the pipette a little above the mark and holds it there by placing a finger on the top of the pipette. Carefully releasing the pressure of the finger, he allows the milk to run slowly out of the pipette until the top of the milk coincides with the mark on the pipette. Then the delivery tube of the pipette is inserted through the neck of the test bottle, and the

finger removed to allow the milk to run into the bottle. The drop remaining in the pipette is blown out, and the pipette removed.

The test bottle should be marked with a pencil to identify the samples; and it is always desirable to test each sample in duplicate.

The amount of acid used for each test bottle is 17.5 ml (fig. 3, *C*), and the acid should be of about the same temperature as the milk to which

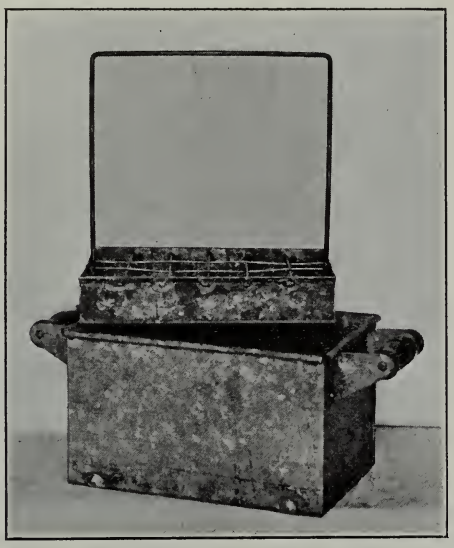

Fig. 4.—Type of water bath used for milk and cream samples. (From Cir. 230.)

it is added (between 60° and 70° F). One should tip the test bottle slightly so that the acid will run down the side of the neck and should revolve the test bottle while adding the acid in order to wash down any traces of milk adhering to the bottle neck. The acid and milk are mixed by a smooth, rotary motion until a uniform color is obtained throughout. One should be careful to use the proper motion for mixing and to continue for a sufficient time.

The bottles are then placed in the centrifuge, being arranged around the carrier at equal intervals that will keep it balanced. If necessary, test bottles containing water are used to keep the socket-carrier balanced. They are then centrifuged for 5 minutes after the machine has attained the proper or "normal" speed, after which they are filled with hot soft water (140° F or above) to the base of the neck. The test bottles are centrifuged again for 2 minutes after proper speed has been attained. The fat column is brought within the graduated portion of the neck of the bottle by adding more hot soft water. Then follows a final centrifuging for 1 minute at the proper speed.

The test bottles are now transferred to a water bath (fig. 4) of such depth that the top of the fat column does not come above the level of the water. The temperature of the bath is maintained between 130° and 140° F as long as any bottles are in it. The tests should remain in the

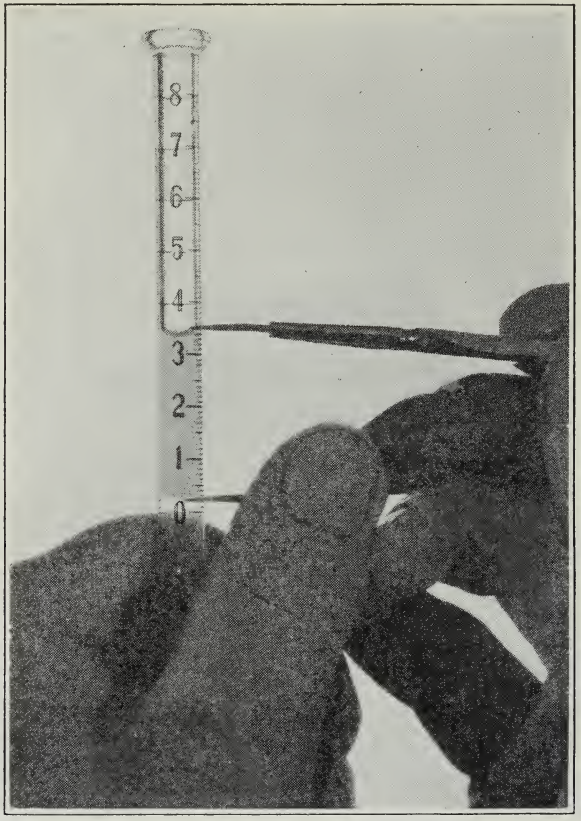

Fig. 5.—The correct position of the hands and dividers when measuring the fat column of milk tests

water at least 10 minutes. Each test must be read immediately upon its removal from the water bath.

The fat column at the time of reading should be translucent, golden yellow or amber, and free from visible suspended particles. All other tests should be rejected, especially in highly accurate work. The reading is taken with a pair of dividers (fig. 5), one point of the dividers being placed at the extreme bottom of the fat column and the other point at the top of the upper curved surface. Then, without changing the spread of the dividers, one point is placed on the zero mark of the graduations;

the other point then indicates the percentage of fat in the milk. This reading should be recorded immediately.

The bottles should then be emptied into a special jar (fig. 6) because this waste acid is still very corrosive and must not be emptied into ordinary drains or where animals can contact the soil that is saturated with

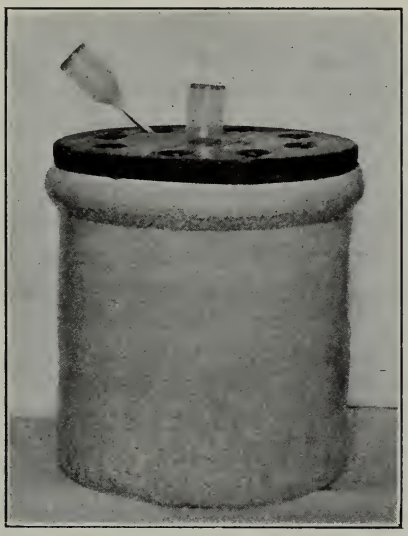

Fig. 6.—Convenient jar for receiving waste acid. (From Cir. 230.)

it. If the bottles are vigorously shaken while being emptied, the sediment in the bottom is easily removed, and the bottles are more easily cleaned by the procedure described on page 1.

IMPERFECT TESTS

Dark-colored fat columns containing black specks may be caused by too strong acid, by too much acid, by too high a temperature of either milk or acid before they are mixed together, by adding the acid directly into the milk instead of allowing it to run down the side of the neck of the bottle, by allowing the acid and milk to stand in the bottle too long before mixing, or by not using a smooth rotary motion when mixing the acid and milk together.

Pale-colored fat columns containing white specks may be caused by weak acid, too little acid, low temperatures of either acid or milk before they are mixed together, or insufficient mixing.

Bubbles appearing in the fat column are caused by using hard water to fill the bottles between the centrifuging periods.

FAT IN CREAM

The Babcock method is also used in testing cream, but a few changes in the procedure and equipment are necessary. Cream varies so greatly in its specific gravity that a pipette does not always deliver the same weight. Also the amount of cream sticking to the sides of the pipette varies so much that a given weight of cream cannot be delivered from the pipette. Air bubbles are frequently trapped in the cream, making it impossible to fill the pipette properly. All these difficulties can be overcome by weighing the cream into the test bottle instead of measuring it as with milk.

Cream Balances.—These balances range in capacity from 1 to 12 bottles, and the 4-bottle torsion balance is very satisfactory. The balance should be set on a level, solid foundation, should be protected from even the slightest draft, and should be checked for accuracy at regular intervals. Being very sensitive, the balance must be handled carefully and preferably not carried or moved. The operator should stand directly in front of it and should *keep the beam locked at all times except during the brief moment necessary to observe whether it is in balance.* When properly balanced, the pointer will swing one or two divisions equally on each side of center without coming to rest.

Cream Test Bottles.[7]—Two cream test bottles are accepted for official work in California. They are 9 inches high and are graduated to read from 0 to 50 per cent, the smaller divisions representing 0.5 per cent each. One bottle is designed to read directly the percentage of fat when 18 grams of cream are used; the other when 9 grams of cream are used.

Procedure in Testing Cream for Fat.—Cream samples are taken in the manner described for sampling on page 2, although greater care must be used because the viscosity of cream makes a uniform mixture more difficult to secure.

The sample is prepared by warming to between 90° and 110° F in order to facilitate mixing and pipetting. Avoid heating the sample too fast or too long or allowing the warm cream to stand too long, since these errors will permit melted fat to separate and will make it impossible to mix the sample properly. The warmed sample is mixed thoroughly by pouring from one container to another three or four times.

Test bottles are placed on the balance (fig. 7) and balanced by the tare weight. Then the weight (9 or 18 grams according to the test bottle used) is placed on one pan, and cream is introduced by means of a pipette into the test bottle on the opposite pan. If too much cream is added, a small portion may be poured from the test bottle, and the bottle returned to the balance. Experience will enable the operator to check

[7] See footnote 5.

the flow of cream from the pipette when the correct charge has been delivered. Care must be used to get all the cream into the test bottle and not on the outside or on the pan of the balance.

The amount of acid to be added varies with the amount of cream in the test bottle and the richness of the cream. Usually from 4 to 8 ml of

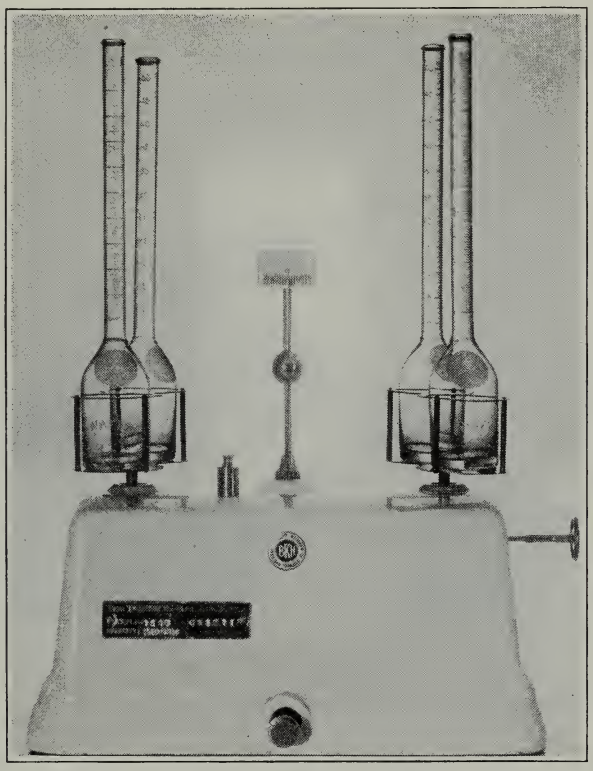

Fig. 7.—Cream balance, weight, and test bottles.

acid are required for the 18-gram charge. The amount of acid necessary will decrease as the richness of the cream increases, because there are less solids-not-fat to be dissolved and less moisture to weaken the acid. Since the richness of the sample cannot be foretold, the operator must learn to add sufficient acid to give a coffee-brown color after mixing the acid and cream. A darker color indicates that too much acid has been added. When adding the acid, revolve the bottle to wash down the cream adhering to the sides of the neck.

The manner of mixing the acid and cream and of centrifuging the cream tests is exactly the same as described for milk on page 7.

The reading of cream tests, however, differs from the reading for milk. After the tests have been in the water bath at 130° to 140° F for at least 10 minutes and just before the test is read, a few drops of glymol (1 quart of a white mineral oil colored red with 1 ounce of alkanet root or with oil-soluble aniline dye) are allowed to run down the side of the neck of the test bottle. This glymol straightens out the upper meniscus or crescent-shaped curve of the fat column, making possible a more accu-

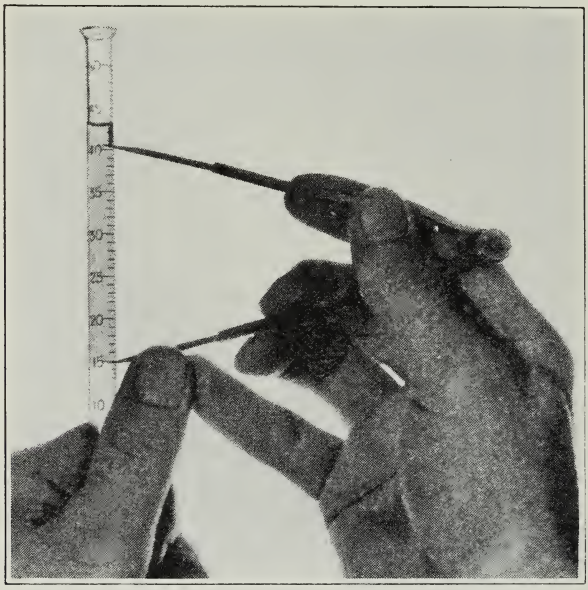

Fig. 8.—The correct position of the hands and dividers in measuring the fat column of cream tests. Note the glymol above the upper point of the dividers.

rate reading. As shown in figure 8, one point of the dividers is then placed at the extreme bottom of the fat column, and the other point adjusted to the straight line at the bottom of the glymol. Without changing the spread of the dividers, one point is placed on the zero mark on the neck of the bottle; and the percentage of fat is read where the other point meets the scale.

The test bottles should be shaken while being emptied into the waste-acid jar as directed on page 9 and then thoroughly cleaned as described on page 1.

Imperfect tests and their causes are described on page 9.

FAT IN SKIM MILK

The Babcock method of testing skim milk requires a double-neck skim-milk test bottle (fig. 3, *A*) having one large neck for introducing the

charge and one neck with a fine bore and graduations sufficiently small to record 0.01 per cent fat.

The directions outlined for testing milk should be followed with a few exceptions. One should add slightly more acid and add it in two portions, mixing thoroughly after each portion is added.

When skim-milk test bottles are being placed in the centrifuge, the larger neck should be toward the center. The tests are centrifuged for 10, 3, and 2 minutes, water being added between each period as in testing milk.

The glassware should be cleaned as described on page 1.

ACIDITY IN MILK AND SKIM MILK

Mann's acidity test is frequently used to determine the acidity of milk and its products. This test is based upon the use of a dilute alkali to neutralize the acidity and of an indicator to show the end-point of the titration.

Alkali Solution.—This solution must be of the proper strength, designated as one-tenth normal. It may be prepared by accurately diluting the concentrated solution sold by dairy-supply companies, observing the directions on the bottle of concentrated solution and being careful to rinse all the concentrated solution thoroughly from the bottle. If the necessary equipment is available the solution may also be prepared by dissolving 4 grams of sodium hydroxide in distilled water and adding sufficient distilled water to make 1,000 ml of solution.

The Burette.—The amount of alkali used in the acidity test is conveniently measured from a burette graduated so that each small division represents 0.1 ml. The burette shown in figure 9 is attached to the bottle of alkali for convenience in filling. The tip of the burette below the pinchcock must first be filled with alkali free from bubbles.

Procedure in Determining Acidity of Milk.—The sampling should be done as described on page 2. The sample is prepared and pipetted in the manner described on page 5, the 17.6 ml pipette being used to transfer 18 grams of milk into a white porcelain cup. Then 2 to 4 drops of the indicator solution (a 3⅓ per cent solution of phenolphthalein in 90 per cent ethyl alcohol) are added to the cup.

After making sure that no bubbles of air are trapped in the tip of the burette, add the alkali solution drop by drop. The sample in the white cup is stirred with a glass rod to mix each drop of alkali thoroughly as it is added. This process is continued until the sample shows the very faintest pink color or until a definite change in its color remains after mixing for one-half minute without further additions of alkali. Extreme care is necessary in detecting this point.

The reading of the burette before starting the test is subtracted from the reading at the end of the test, to obtain the number of milliliters of alkali solution added to the cup. The resultant figure (the number of

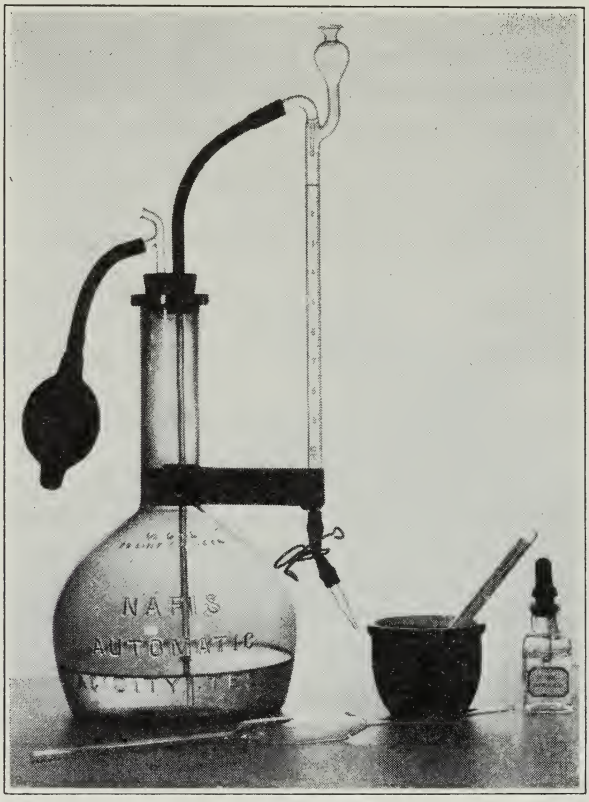

Fig. 9.—Equipment for making Mann's acidity test.

milliliters of alkali added to the cup) is divided by 20 to obtain the percentage of acidity in the sample.

ACIDITY IN CREAM

Mann's acidity test, described in the preceding section, is also used, with a few minor changes, for testing the acidity of cream. A 9 ml pipette is used to transfer 9 grams of the cream to the white cup, and then the pipette is filled with warm distilled water to rinse out the cream adhering to the inside of the pipette. This 9 ml of rinse water is added to the cream in the white cup.

The indicator and alkali are added in the same manner as for milk, and the number of milliliters of alkali used is determined.

The percentage of acidity in the cream is calculated by dividing by 10 the number of milliliters of alkali used.

SPECIFIC GRAVITY AND SOLIDS IN MILK AND SKIM MILK

A special type of hydrometer, known as a lactometer, is used to determine the specific gravity and percentage of solids-not-fat in milk. The latter, when determined by this method, may be in error by as much as 0.3 per cent unless the operator is skilled and observes extreme care in the procedure. The most important reason for this variation in accuracy is that the milk sample may differ widely (both in composition and behavior) from the "average" used in determining the empirical formula for calculating the results.

The method is based upon the principle that a freely floating body displaces a quantity of liquid weighing the same as the floating body and that the density of a solution depends upon its total solids content. In the case of milk, the fat and the part called solids-not-fat constitute the total solids.

The Lactometer.—Of the several types of lactometers on the market, the most acceptable is graduated in "Quevenne degrees" with the small divisions representing one-tenth of a degree. Many technicians prefer the large lactometer recommended by the Bureau of Dairy Industry of the United States Department of Agriculture, but about a pint of milk is required to float it properly.

Lactometers are graduated on the stem, reading from the top down. A scale reading from 27 to 40 is a convenient range. Since milk having less solids will cause the lactometer to sink lower into the milk, the lower readings on the scale must be at the top of the stem.

Procedure in Determining Specific Gravity and Solids in Milk.—The sample is taken as directed on page 2. It must be kept cold (40° to 50° F) for one or preferably more hours and then placed in cool water, which will slowly raise the temperature to between 55° and 65°, preferably to 60°. An hour or longer should be required to raise the temperature to 60°.

The milk is thoroughly mixed by pouring from one container to another until a homogeneous mixture is obtained. Care should be taken that these containers do not change the temperature of the milk.

The milk is then poured into a cylinder of the same temperature as the milk. This cylinder should have a diameter at least 1 inch greater than the largest diameter of the lactometer, together with a capacity that will float the lactometer. The cylinder should be filled to such a point that when the lactometer is placed in the milk the cylinder will overflow.

The lactometer is immediately placed in the milk and pushed down

to wet its stem. Care should be used to have it float freely, not near the sides of the cylinder.

The lactometer should be allowed to stand at least one-half minute and not more than two minutes before reading. All bubbles should be blown

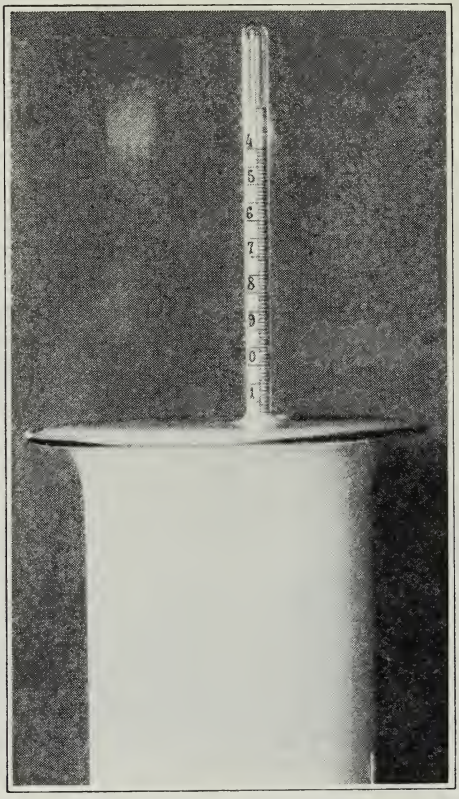

Fig. 10.—The lactometer reading taken to the nearest small division at the top of the meniscus is 31.3. In the illustration above, the instrument is turned slightly and does not show the full figure.

from the surface, particularly around the stem. The lactometer (fig. 10) is read to the nearest small division on the scale, at the top of the meniscus on the stem, the eye being kept at the same level as the meniscus.

If the temperature of the milk is not exactly 60° F, the lactometer reading must be "corrected" for temperature. This correction is made by adding 0.1 for each degree above 60° F to the observed lactometer reading or by subtracting 0.1 for each degree below 60° F from the reading, provided the lactometer is graduated in Quevenne degrees.

The specific gravity and the percentage solids-not-fat may be calculated by substituting in, and solving the following formulas. Where L is the "corrected" lactometer reading (that is, the lactometer reading corrected for temperature) and F is the fat test obtained by the Babcock method and expressed as per cent (that is, if the milk tests 3.8 per cent) then $F = 3.8$.

$$\frac{L}{1{,}000} + 1 = \text{specific gravity.}$$

$$\frac{L}{4} + \frac{F}{5} = \text{per cent solids-not-fat.}$$

SEDIMENT IN MILK

The sediment test is a simple method of determining the amount of dirt or insoluble foreign material in milk. It is based upon the ability of a cotton pad to remove very small particles so that they may be seen.

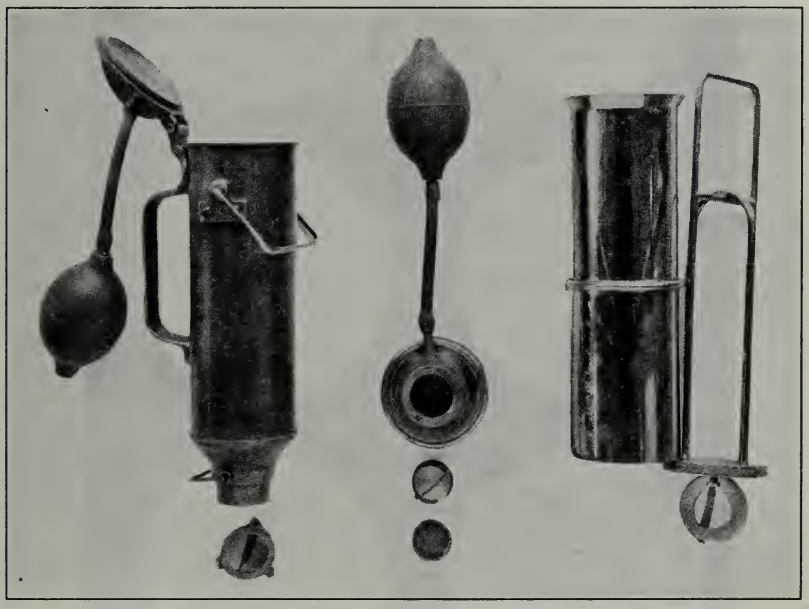

Fig. 11.—Some of the types of sediment testers. (From Cir. 327.)

The several sediment testers available on the market all give satisfactory results when properly used. Three types are shown in figure 11. Instructions accompany each tester when purchased and therefore will not be repeated here. One should, however, observe certain precautions not mentioned by the manufacturers.

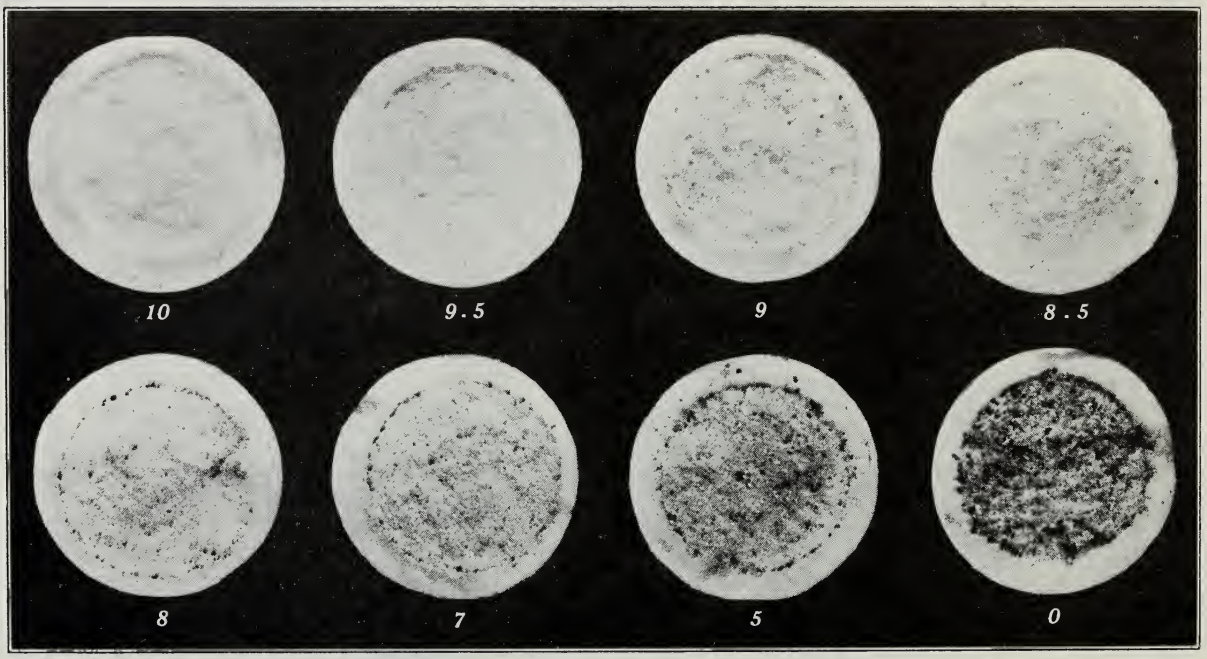

Fig. 12.—Standard series of prepared disks, actual size. (From Cir. 327.)

The sample should be taken as directed on page 2. Since exactly one pint of milk is used in the test, a quart should be taken for a sample. The sample is then thoroughly mixed by pouring from one container to another three or four times until a homogeneous mixture is obtained. If necessary, the sample should be warmed to 100° F in order to soften lumps of cream.

When the cotton disk is fitted into the tester, exactly one pint of the prepared sample is forced through the disk. Care must be used to get all the milk to pass through the disk and not around it. Especial care should be used to have the last teaspoonful of milk pass through, since this milk will otherwise rinse away much of the sediment when the operator tries to remove the disk from the tester.

The sample of milk is given a numerical rating for sediment by comparing the appearance of the cotton disk with a standard series of prepared disks shown in figure 12.

SEDIMENT IN CREAM

A sediment test of cream is made in the same manner as the sediment test in milk. Only 4 ounces of cream are used, however, instead of 1 pint as in milk. Also the cream is diluted with about ½ pint of boiling water, free from even small particles of dirt.

If the cream is sour, the boiling water should contain a small amount of alkali such as baking soda. A convenient alkali solution for sour cream is made by dissolving 2 tablespoonfuls of baking soda in 3 quarts of water and passing it through a cotton disk to remove any sediment before heating it for use in diluting the cream.

15m-5,'37